Biochemical Oxygen Demand Audit Checklist * (BOD and CBOD)

Based on NR 219 (2004), NR 149 (1998) and Standard Methods 5210 B (18th, 19th and 20th Editions)

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	Sample Storage and Pretreatment	Y	N	Notes	Citation
1	Are BOD samples set up within 2 hours or stored at ≤ 6 ºC prior to analysis?				NR 219; Table F
2	Are samples set up within hold time (≤48 hours)?				NR 219; Table F; 5210 B
3	Are samples checked for residual chlorine?				5210 B; 4.e.(2)
4	If residual chlorine is found is the sample neutralized?				5210 B; 4.e.(2)
5	Is the pH of samples checked prior to set up?				20th 5210B; 4.e.
6	Are samples pH adjusted to pH 6.5 - 7.5 (if not in pH 6.0 - 8.5 initially)?				20th 5210B; 4.e.
7	If pH adjustment is done is the amount of acid or base used limited to ≤ 0.5% of sample volume?				5210B; 4.e.(1)
8	Are samples warmed to 20 +/-3 ºC before analysis? (18th and 19th say 20 +/-1 ºC)				20th 5210B; 1.b. 18th/19th 5210B; 4.e.(5)
9	Are samples over the 100% DO saturation value identified and treated for super saturation?				5210B; 4.e.(4)

	Equipment	Y	N	Notes	Citation
10	Are all necessary reagents and glassware available? Reagents purchased_____or prepared_____?				5210 B; 2.& 3.
11	Is the DO meter properly calibrated on each analysis day? Water sat.air_____ Air sat. water____ or Winkler_____				NR 149.14 (3)a.
12	Does the incubator maintain samples at 20 +/- 1 ºC during the 5 day test period?				5210B; 2.b.
13	Is the room temperature sufficiently controlled to meet the test requirements of 20+/- 3 ºC?				20th 5210B; 4.
14	Is the room temperature sufficiently controlled to meet the test requirements of 20 +/- 1ºC?				18th/19 5210B; 4.

	Sample Seeding	Y	N	Notes	Citation
15	What is the seed source and which samples are seeded?	NA	NA		5210B; 4.d.
16	Is the seed properly prepared?				5210B; 4.d.(1)
17	Are industrial, disinfected (UV or chlorine), or pH-adjusted samples seeded?				5210B; 4.e.(1&2)
18	Are at least two seed controls run? (should have at least two that meet depletion criteria and recommend a seed correction factor between 0.6 to 1.0 mg/L)				5210B; 4.d.(2)
19	Are seed correction factors properly calculated and used to adjust results of seeded samples?				5210B; 4.d.(2)

Total Suspended Solids (TSS) Checklist *

Based on NR 149 (1998), NR 219 (2004) and Standard Methods (18th, 19th and 20th Editions)

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	Sample Storage and Pretreatment	Y	N	Notes	Citation
1	Are TSS samples stored at ≤ 6 ºC until analyzed?				NR 219; Table F
2	Are samples analyzed within the hold time of 7 days?				NR 219; Table F
3	Is sample well stirred using a magnetic stirrer prior to analysis?				2540D; 3.c.

	Equipment	Y	N	Notes	Citation
4	Are glass fiber filter disks (without organic binder) approved brands or other products that give equivalent results?				2540 D; 2. 2540C; 2a.
5	Does the lab have an analytical balance capable of weighing to 0.1 mg?				2540 D; 2. 2540C; 2f.
6	Does the lab take steps to verify the accuracy of the analytical balance?				NR 149.14 (3)a.
7	Is a suitable desiccator with color indicating desiccant or an instrumental indicator used?				2540 D; 2. 2540C; 2d.
8	Is the drying oven capable of maintaining 103 - 105 ºC?				2540 D; 2. 2540C; 2e.

	General Procedural Observations	Y	N	Notes	Citation
9	Is the fiber filter disk rinsed with three 20mL portions of reagent-grade water prior to drying?				2540D; 3.a.
10	Is the filter dried at 103-105ºC for at least an hour, cooled in desiccator, and then weighed?				2540D; 3.a.
11	Is the filter re-dried and re-weighed until weight change is less than 4% of the previous weight, or 0.5 mg, whichever is less?				2540D; 3.a.
12	If filter is not re-dried is the filter dried overnight with a quarterly redry done to prove dryness is achieved?				Lab Cert Allowance
13	Is sample size appropriate to yield between 1.0 and 200 mg of residue?				Lab Cert Allowance 2540D; 3.b.
14	Is the sample filtered and then washed with at least three 10mL portions of reagent-grade water?				2540D; 3.c.
15	Are very large particles removed from the sample if it is deemed that they are not representative?				2540D; 1.b.
16	Is the filter with sample dried at 103-105ºC for at least an hour, cooled in desiccator and then weighed?				2540D; 3.c.
17	Is sample re-dried and re-weighed until residue weight change is < 4% of the previous weight, or 0.5 mg, whichever is less?				2540D; 3.c.
18	If sample is not re-dried is it dried overnight with a quarterly redry done to prove dryness is achieved?				Lab Cert Allowance
19	Are calculations of suspended solids done correctly?				2540D; 4.

	General Procedural Observations	Y	N	Notes	Citation
20	Are the proper reagents prepared_____ or purchased_____ for dilution water preparation?				5210B; 3.
21	Are all reagents properly labeled and in good condition?				5210B; 3.
22	Is the dilution water properly made and stored?				5210B; 4.a.& b.
23	For sample dilutions of greater than 1:100 is a preliminary dilution done?				5210 B; 4.f.(2)
24	Are sample volumes adjusted so that depletion criteria are met as often as possible?(d*epletion of > 2 mg/L DO & remainder of > 1 mg/L DO)*				18th/19th 5210B; 4.f.
25	Do at least two sample volumes meet the depletion criteria?				20th 5210 B; 4.f.
26	Are at least two sample dilutions run for each sample?				5210B; 4.f.
27	For samples over 201 mL are additional nutrients added? Are the nutrients powder ____ or liquid____(0.33 mL per 300 mL)?				20th 5210B; 4.f.
28	Are sample bottles water sealed prior to incubation?				20th 5210B; 4.f. 18th/19th 5210B; 2.a.
29	If nitrification inhibitor is used, does the lab have certification or registration for CBOD?				NR 149.04 (1)
30	Are CBOD samples properly labeled and the results reported as CBOD?				149.06 (1)
31	Have sliding BODs been observed?				5210B; 4.e.(3)
32	If sliding BODs have been observed have steps been taken to identify the source of the toxicity?				5210B; 4.e.(3)
33	Are BOD values properly calculated for all samples?				5210B; 5.

	Glucose-Glutamic Acid (GGA) Standard	Y	N	Notes	Citation
34	Is GGA standard properly prepared or commercially purchased?				5210B; 3.h.
35	Is GGA standard analyzed at a 2% dilution (6 mL to 300 mL) using a concentration that yields 3 mg/L glucose and 3 mg/L glutamic acid in the GGA test bottle?				5210B; 4.c.
36	Are GGA standards analyzed after every 20 samples or weekly at a minimum (if < 20 samples are run in a week)?				NR 149.14 (3)(c)4
37	Are seed controls run and correctly applied to GGA data?				5210B; 4.d.(2)
38	Do GGA results meet the 198 +/- 30.5 mg/L BOD standard? *(167.5 - 228.5)* Multiple GGA standards cannot be averaged.				5210B; 6.

	BOD-Specific Quality Control **(refer to the QC and Records checklist for other QC/records requirements)**	**Y**	**N**	**Notes**	**Citation**
39	Do all samples, standards and seed controls used to calculate results meet the depletion criteria?				5210B; 5.
40	If criteria are not met are data excluded from calculations or qualified if there are no acceptable dilutions to use?				5210B; 5.
41	Is a dilution water blank run with each batch of samples and/or batch of dilution water?				5210B; 4.h.
42	Do dilution water blanks meet the depletion limit of < 0.2 mg/L DO?				5210B; 4.h.

Other Observations

Ammonia Ion-Selective Electrode (ISE) Method Checklist *

Based on NR 149 (1998), NR 219 (2004), EPA Method 350.3, and Standard Methods (18th, 19th and 20th editions)

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	Sample Storage and Pretreatment	Y	N	Notes	Citation
1	Are ammonia samples stored at ≤ 6ºC prior to analysis?				NR 219; Table F
2	If analysis is not started immediately (≤ 15 minutes) is the sample preserved at pH of <2 using sulfuric acid?				NR 219; Table F
3	Are samples analyzed within the hold time of 28 days (stored at pH <2 and ≤ 6ºC)?				NR 219; Table F
4	Are samples and standards at room temperature before analysis starts?				19 & 20:4500 NH3 - D; 4b. 18:4500 NH3 - F; 4b.
5	Are samples distilled unless they are wastewater samples and a copy of the SLH distillation study is on file?				Lab Cert Allowance NR 219 Table B
6	If sample concentration is above the top standard is the sample diluted and re-analyzed?				4500 NH3-B; 4.e.

	Equipment	Y	N	Notes	Citation
7	Are all the necessary apparatus, reagents and glassware available?				19&20: 4500-NH3-D; 2&3. 18: 4500 NH3 - F; 2 & 3 350.3 (5&6)
8	Is ammonia -free water being used for analysis?				4500-NH3-B;3.a. 350.3 (6.0)
9	Is the ammonia probe maintained properly? *Including regular membrane changes, filling solution changes, proper storage (in 100 or 1000 ppm std.), etc.?*				NR 149.14 (3)a.

	Calibration/Sample Measurement	Y	N	Notes	Citation
10	Is 100 mLs of sample/standard used to do the analysis?				4500 NH3-B; 4.e.
11	Are samples slowly stirred using a thermally insulated magnetic stirrer?				4500 NH3-B; 4.b.
12	Is the NaOH buffer solution added to samples and standards after the probe is immersed in the sample?				19&20:4500 NH3-D; 4b 18:4500 NH3-F;4b. 350.3 (7.2)
13	Is enough buffer used to raise the sample pH above 11?				19&20:4500 NH3-D; 4b. 18:4500 NH3-F;4b. 350.3 (7.2)
14	If more than 1mL of buffer is added to samples (and less is added to the calibration standards) is the amount noted and used in the calculation?				19&20:4500 NH3-D; 4b. 18:4500 NH3-F;5.
15	Is the meter allowed sufficient time to stabilize (about 3 minutes on the low level standards/samples)?				4500 NH3-B; 4.b.& e.
16	Is the meter calibrated daily with at least 3 standards?				NR 149.14 (3)b.
17	Is the meter calibrated starting with the lowest standard and progressing to higher standards?				19&20:4500 NH3-D; 4b. 18:4500 NH3-F;4b. 350.3 (7.2)
17	Does the calibration yield a slope of 59 mV (54-60) or slope required by meter manufacturer? Orion -54 to -60 mV, Corning -50 to -60 mV, Acumet -55 to -63 mV Hach -54 to –62 mV				19&20:4500 NH3-D; 4c. 18:4500 NH3-F;4c.

Total Phosphorus - Ascorbic Acid Method Checklist *

Based on NR 219 (2004), NR 149 (1998), HACH 8190 and Standard Methods (18th, 19th and 20th Editions)

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	Sample Storage and Pretreatment	Y	N	Notes	Citation
1	Are phosphorus samples stored at ≤ 6ºC prior to analysis?				NR 219; Table F
2	If analysis is not started immediately (≤ 15 minutes) is the sample preserved at pH of <2 using sulfuric acid?				NR 219; Table F
3	Are samples analyzed within the hold time of 28 days (preserved at pH <2 and ≤ 6ºC)?				NR 219; Table F

	Equipment	Y	N		Citation
4	Are all the necessary apparatus, reagents and glassware available?				4500-P-E; 2&3.
5	Does glassware cleaning include a hydrochloric acid rinse or is clean glassware stored filled with distilled water and subjected to occasional acid rinse?				4500-P-E; 2.
6	Is combined reagent made up fresh daily and used within 4 hours?				4500 P-E; 3.

	Standard Persulfate Digestion	Y	N		Citation
7	What is digested? _____ samples _____standards _____blanks	NA	NA		NA
8	Are reagent to sample volumes proportional to those required by the method?				4500-P-B(5)c.
9	Is mixed sample measured into a flask, then phenolphthalein indicator added? If red color appears is sulfuric acid solution added until it clears?				4500-P-B(5)c.
10	Then is sulfuric acid solution and either ammonium persulfate or potassium persulfate added to the flask?				4500-P-B(5)c.
11	Are samples gently boiled on a hot plate for 30-40 minutes or until the volume is 10 mL?				4500-P-B(5)c.
12	Alternatively, are samples heated for 30 minutes in an autoclave or pressure cooker at 98 to 137 kPa?				4500-P-B(5)c.
13	Is the sample then cooled, diluted with distilled water, phenolphthalein added and then neutralized with NaOH to a faint pink color?				4500-P-B(5)c.
14	Is digested sample pipetted into a flask, phenolphthalein indicator added, and then if red 5N sulfuric acid added until the solution is clear?				4500-P-E 4a.
15	Is combined reagent then added and mixed?				4500-P-E 4a.

	HACH Persulfate Digestion	Y	N		Citation
16	If samples are preserved prior to analysis, are they warmed to room temperature and adjusted to pH 7 before beginning digestion (record volumes of base added)?				HACH 8190
17	Is 5 mL of neutral sample added to a total phosphorus test vial (already contains acid) and then one potassium persulfate pillow is added to the vial?				HACH 8190
18	Vial is capped tightly and shaken, then placed in the pre-heated block digester at 150 ºC for 30 minutes?				HACH 8190
19	Is the sample then cooled, 2 mL of 1.54N sodium hydroxide standard solution added to the vial, and then capped and shaken?				HACH 8190
20	Is the contents of one phosver3 phosphate pillow then added to the vial, capped, shaken and 2 minute timer is started?				HACH 8190

	Calibration/Sample Measurement	Y	N		Citation
21	Is the spectrophotometer calibrated with at least 3 standards and a blank?				NR 149.14 (3)b. 4500-P-E; 4c.
22	Is the calibration curve acceptable (0.995 or better), and properly used to derive sample concentrations based on absorbance data?				NR 149.14
23	Are samples above the upper end of the calibration curve diluted and re-analyzed?				
24	For samples digested using autoclave/hotplate Is the sample absorbance measured between 10 and 30 minutes after the addition of the combined reagent?				4500-P-E 4a.
25	For samples digested following HACH method is the vial wiped clean and the absorbance measured between 2 and 8 minutes after phosver3 pillow packet was added?				HACH 8190
26	Is absorbance measured at the appropriate wavelength - (610, 660, 880, or 890 depending on the method)?				4500-P-E HACH 8190
27	Is a blank run with each sample batch and processed in the same manner as the samples?				NR 149.14 (3)d.
28	Is a known standard analyzed on any day that a curve is not generated?				NR149.14; 3b., 4500-P-E 4c.
29	Is the correct criteria applied to the known standard (+/- 10%)?				NR149.14; 3b., 4500-P-E 4c.

	Other Observations

Quality Control (QC) and Record Requirements Checklist *
for BOD, TSS, Ammonia and Phosphorus

Based on NR 149 (1998). Indicate yes (Y), no (N) or not applicable (NA) for each analyte. SC indicates that you should see checklist (SC) that is specific to that analyte.

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	Quality Control	BOD	TSS	NH3	P	Notes	Citation
1	Is a method blank analyzed with each batch of samples?	SC	NA				NR 149.14(3)d.
2	Is corrective action taken if the method blank is above the LOD, 5% of regulatory limit or 5% of the sample concentration (whichever is highest)?	SC	NA				NR 149.14(3)d.
3	Is a replicate run after the analysis of 20 samples of each matrix type (at least 1 replicate for 20 samples)?						NR 149.14 (3)e.
4	Are quality control (QC) limits for replicates calculated for each matrix (unless lab has < 20 QC results/year then they can set QC limits)?						NR 149.14 (3)g.
5	Are QC limits used to assess replicate performance each time replicates are analyzed?						NR 149.14 (3)g.
6	Is a spike run after the analysis of 20 samples of each matrix type (at least 1 spike for every 20 samples)?	NA	NA				NR 149.14 (3)f.2
7	Are QC limits for spikes calculated for each matrix (unless lab has < 20 QC results/year then they can set QC limits)?	NA	NA				NR 149.14 (3)g.
8	Are QC limits used to assess spike performance each time spikes are analyzed?	NA	NA				NR 149.14 (3)g.
9	Is a known standard analyzed after 20 samples (+/-10%)?	SC	NA				NR 149.14 (3)c.
10	When QC limits for standards, replicates, spikes or blanks are exceeded is corrective action taken?						NR 149.14 (3)h.
11	Are blind standards analyzed three times a year with 3 to 5 month spacing between each set?						NR 149.14 3(j).
12	When a blind standard result fails is a new standard ordered and analyzed after taking corrective action?						NR 149.14 3(j).

See next page for records requirements.

	Records	BOD	TSS	NH3	P	Notes	Citation
13	Are all records available for last 3 years of analysis?						NR 149.06 (1)
14	Are records kept in secure manner, recorded in ink or stored electronically w/ safeguards?						NR 149.06 (5)
15	Are sample results traceable to analyst, date collected, and method used including raw data, calculations, results and final report?						NR 149.06(1)a.
16	Are sample collection records complete? (i.e. sample dates, location, sampler, sample condition, preservation etc.)						NR 149.06 (1)
17	Is the raw data (i.e. absorbance, millivolts) recorded for all samples and standards?						NR 149.06(1)a.
18	Are sample results clearly traceable to the calibration curve that was used to generate them?						NR 149.06(1)a.
19	Are equipment maintenance records for all analytical equipment kept?						NR 149.06 (1)
20	Are clear records of replicates and associated control limits available and current?						NR 149.06 (1)
21	Are clear records of spikes and associated control limits available and current?	NA	NA				NR 149.06 (1)
22	Are records associated with blind and reference samples available?						NR 149.06 (1)
23	Are records of corrective actions taken in response to QC failures available?						NR 149.06 (1)
24	Does corrective action include qualification of data on data report or DMR?						NR 149.14 (3)h.

	Other Observations